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A
FORCED VIBRATION NON-RESONANT METHOD
FOR THE DETERMINATION OF
COMPLEX MODULUS IN THE
AUDIO FREQUENCY RANGE

J. P. Szabo

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Abstract

A forced-vibration, non-resonant method for the determination of the dynamic mechanical properties of elastomeric materials in the audio frequency range was developed and evaluated using a commercial damping material, Isodamp C-1002 from EAR Specialty Composities. Measurements of storage modulus and loss factor were made in the 11 Hz to 10 kHz range on the damping material using compressive and tensile excitation. The elastomer had a loss factor near 1.0 and a storage modulus which varied between 6×10^6 Pa and 3×10^8 Pa in the frequency range studied. For tensile excitation, the error in the calculated modulus was low up to the resonance frequency of the sample, usually ~1700 Hz. For compressive excitation of thin sheets, the error was greatest at the low and high ends of the frequency range.

Résumé

Une méthode exploitant des vibrations forcées non résonantes pour déterminer les propriétés méchaniques dynamiques des élastomèrès dans la gamme des fréquences audibles a été mise au point et évaluée au moyen d'un matériau amortisseur commercial, l'Isodamp C-1002 d'EAR Specialty Composites. Le module de conservation et le facteur de pertes ont été mesurés, sur l'intervalle de 11 Hz à 10 kHz, dans le matéteriau amortisseur soumis à des contraintes tantôt de compression, tantôt de traction. L'élastomère a présenté un facteur de pertes de près de 1, 0 et un module de conservation variant entre 6×10^6 et 3×10^8 Pa dans la gamme de fréquence donné. En Traction, l'erreur sur le module calculé a été faible jusqu'à la fréquence de résonance de l'éprouvette, normalement ~1700 Hz environ. En compression, l'erreur pour des feuilles minces a été la plus élevée aux valeurs maximale et minimale de la gamme de fréquences.

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Introduction

The unique properties of polymers make them ideally suited for damping structure-borne vibrations when used in anti-vibration mounts or sheet damping applications, and in reducing air or water-borne sound when used in anechoic coatings[1]. The frequency and temperature dependence of the viscoelastic properties of polymers is essential information for the selection and design of materials for noise and vibration damping. There are a variety of commercially available instruments, known as Dynamic Mechanical Thermal Analyzers (DMTA), that are able to measure the complex modulus of polymers over a wide temperature range (-100 °C to 500 °C); however, the frequency range for these instruments is limited to lower frequencies, typically 10^{-2} Hz to 10 Hz. If data at higher frequencies are required, then an apparatus is usually constructed, consisting of a rig to clamp the sample, transducers to measure force and displacement, and electronics to process the signals.

An important consideration in the design of a complex modulus apparatus is the excitation technique. Resonance methods, in which at least one end of a sample can freely move, are not well suited to high loss, low stiffness samples[2] and data is usually analyzed at only a few frequencies corresponding to the resonance frequencies of the sample. Forced vibration non-resonant excitation, in which the sample is clamped at one end and driven at the other, is well suited to low stiffness samples and yields data over a continuous range of frequencies for a given sample.

Another important aspect of the excitation technique is the mode of excitation. Samples may be excited in compression, tension, torsion, shear, or flexure. The choice of excitation mode depends on the stiffness of the sample, and whether shear, Young's (extensional), longitudinal, or bulk modulus is of most interest. For elastomers in their rubbery state, shear or tensile excitation works well, but stiffer samples may require a bending mode. A major disadvantage in a bending experiment is that the Poisson's ratio of the polymer, which can vary from 0.3 to 0.5, should be known in order to accurately calculate shear and Young's moduli.

In this paper, the forced-vibration non-resonant excitation method is described in detail for two experimental configurations.

Experimental Procedure

Measurements were carried out on a commercial damping polymer, Isodamp C-1002 from EAR Specialty Composites. Soxhlet extraction of this polymer with acetone

showed it to contain about 50 wt% liquid plasticizer. Pyrolysis/Gas Chromatography-Mass Spectrometry (GC-MS) and Infrared (IR) analysis identified the plasticizer as dioctylphthalate and the deplasticized polymer as polyvinyl chloride (PVC), or a PVC copolymer. Powder x-ray diffraction and Energy Dispersive X-ray Analysis (EDXA) of the polymer revealed that the polymer contained TiO_2 as an inorganic filler.

The complex Young's modulus of the EAR material was measured using two configurations:

<u>Configuration I</u> Figures 1 - 4 illustrate the experimental set up for tensile excitation of a rectangular polymer sample (3.08 mm x 11.80 mm x 26.61 mm). The force transducer (B & K 8200) was bolted into a massive copper plate (5 cm x 38 cm x 71 cm) that served as a rigid support for this system. The top and bottom of the sample were held in place by hand-tightened clamps. The top clamp remained stationary, while the bottom clamp was driven with broadband excitation provided by a B & K 4809 Vibration Exciter, which was supported by an adjustable lab jack. The motion of the driven end of the sample was monitored by an accelerometer (B & K 4374). The force (F) and acceleration ($\ddot{x}$) signals were amplified by charge amplifiers (B & K 2635) before being frequency analyzed by a digital filter analyzer (B & K 2133) in 1/12 octave mode. Both autospectra and cross spectra were collected to obtain the magnitudes $\bar{\bar{x}}$ and $\bar{F}$, as well as the phase angle (φ) between them. The spectral data were transferred over an IEEE bus to a Jemini 286 computer. Calculation of storage moduli and loss factor were carried out using Equations 6 and 8 (see Results and Discussion).

<u>Configuration II</u> Figures 5 and 6 illustrate the experimental set up for compressive excitation of a thin sheet (3.03 mm). The force transducer was bolted directly to the shaker and to a 15 mm x 16 mm aluminum block that served to transmit the force from the vibration exciter to the sample, which was compressed between the aluminum block and the rigid support. Two accelerometers were used to monitor the motion of the vibration exciter (and aluminum block) and the motion of the nominally rigid support. As shown in Figure 6, the amplified accelerometer signals were differenced by a differential ac amplifier (Brookdeal 9454). The net acceleration ($\ddot{y}$) and force (F) signals were then frequency analyzed in 1/12 octave mode. Both autospectra and cross spectra were collected to obtain the magnitudes $\bar{\bar{y}}$ and $\bar{F}$, as well as the phase angle (φ) between them. Calculation of storage moduli and loss factor were carried out using Equations 18 and 20 (see Results and

Discussion). The spectra for this configuration were smoothed using a second order nine point Savitzky-Golay algorithm[3]. There was no need to smooth data from Configuration I.

Results and Discussion

<u>Configuration I</u> Figures 7 and 8 show the measured force ($\bar{F}$), acceleration ($\bar{x}$), and phase angle (φ) for an elastomer sample excited in tension. Notice that the phase angle is a smooth function until the first resonance peak occurs at ~ 1700 Hz. In contrast, the phase angle for Configuration II was well behaved at high frequency, but poorly behaved at frequencies below 100 Hz. For Configuration I, the force transducer was stationary and the only force acting on the transducer was from tensile excitation of the sample. Since the strain on the sample was small[4] (< 0.4 %), the force (F) is proportional to the displacement (x) as described by Hooke's Law:

$$F = kx \tag{1}$$

where k is the sample stiffness. F and x are time (t) varying quantities and thus have amplitude and phase:

$$x = \bar{x}\exp[i\omega t] \tag{2}$$

$$F = \bar{F}\exp[i(\omega t + \varphi)] \tag{3}$$

In the above equation, the barred symbols represent amplitudes of complex variables, $\omega = 2\pi f$ is the angular frequency, φ is the measured phase angle between F and x, and $i = \sqrt{-1}$. Substituting [2] and [3] into [1] gives

$$k = (\bar{F}/\bar{x})\exp[i\varphi] \tag{4}$$

For a tensile experiment on a sample of length L and cross sectional area A, the Young's modulus is defined[2] to be E = k(L/A). Noting from [2] that $\ddot{x} = -\omega^2 x$, it follows that

$$E = -\frac{L}{A}\left\{\frac{\bar{F}}{\ddot{x}}\right\}\omega^2 \exp[i\varphi] \tag{5}$$

The real and imaginary parts of the complex modulus, or storage and loss modulus, are

$$E' = -\frac{L}{A}\left\{\frac{\bar{F}}{\ddot{x}}\right\}\omega^2 \cos\varphi \tag{6}$$

$$E'' = -\frac{L}{A}\left\{\frac{\bar{F}}{\bar{x}}\right\}\omega^2 \sin\varphi \qquad [7]$$

The material loss factor, η, is related to the phase angle between stress and strain on the sample, δ:

$$\eta = \tan\delta = \frac{E''}{E'} = \tan\varphi \qquad [8]$$

In this case the measured phase angle, φ, is identical with δ, the angle between stress and strain.

The storage moduli and loss factors (Figures 9A and 10A) were calculated by Equations 6 and 8, using data shown in Figures 7A, 7C, and 8A. In this configuration, the resonance at 1700 Hz results in large errors in the calculated storage moduli and loss factors at frequencies approaching resonance and above resonance. The modulus increases monotonically with frequency, with rubber-like values ($<10^7$ Pa) at frequencies up to 100 Hz, and with values characteristic of the glass transition ($\sim 10^8$ Pa) at higher frequencies. The calculated loss factors as a function of frequency forms a smooth curve up to 300 Hz, after which the resonance causes large errors.

Curve B in Figures 9 and 10 represents data from a previous study on Isodamp C-1002 by Sun and Mitchell[5]. The technique used by Sun and Mitchell is a transfer function method with an associated "lumped mass" model. Calculation of moduli with the latter method requires the solution of several non-linear equations for each frequency of interest, in contrast to the method described here, for which post-processing of experimental data is quite straightforward. Agreement between the two techniques is reasonable up to the resonance frequency of the sample. Adjustment of pre-load to near zero values was carried out by adjusting the height of the vibration exciter with the lab jack. Increasing the pre-load resulted in larger calculated moduli, but a more quantitative study of this relationship will be reserved for future experiments in which a static load cell will be incorporated into the system.

Configuration II   For this set up, the use of a second accelerometer was found to give a smoother phase angle curve. Figures 7 and 8 show the measured force ($\bar{F}$), net acceleration ($\ddot{y}$), and phase angle (φ) for an elastomer sheet excited in compression against a rigid support. This type of excitation required larger forces for smaller displacements

than Configuration I, since the stress was across the sample thickness, not length. The low signal/noise ratio may have contributed to errors in the phase angle at frequencies below 100 Hz, which resulted in errors in the calculated moduli (see below).

The force acting on the transducer included the inertial force required to move the aluminum block. Thus, the equation of motion for this system must include a mass term[6]:

$$F = m\ddot{x} + k(x - x_0) \tag{9}$$

where F is the measured force, m is the mass of the sample block, x is the displacement of the sample block, x_0 is the displacement of the support, and k is the stiffness of the sample. With the exception of mass, all variables in [9] are time-varying quantities and thus have an amplitude and phase. If we let $y = x - x_0$ be the net displacement, then y, F, and x_0 may be expressed as

$$y = \bar{y} \exp[i\omega t] \tag{10}$$

$$F = \bar{F} \exp[i(\omega t + \varphi)] \tag{11}$$

$$x_0 = \bar{x}_0 \exp[i(\omega t + \theta)] \tag{12}$$

Substituting $y = x - x_0$ and $\ddot{x} = \ddot{y} + \ddot{x}_0$ into [9] gives

$$F = m\ddot{y} + m\ddot{x}_0 + ky \tag{13}$$

Noting from [10] that $\ddot{y} = -\omega^2 y$,

$$F = m\ddot{y} + m\ddot{x}_0 - \frac{\ddot{y}}{\omega^2} k \tag{14}$$

$$\therefore \quad k = -\frac{F}{\ddot{y}} \omega^2 + \omega^2 m \left(1 + \frac{\ddot{x}_0}{\ddot{y}}\right) \tag{15}$$

If the support is very rigid, then $\ddot{x}_0 \ll \ddot{y}$ and Equation [15] can be approximated[6] by

$$k = -\frac{F}{\ddot{y}} \omega^2 + \omega^2 m \tag{16}$$

The validity of this approximation requires verification by observation of the accelerometer signals. Substituting [10] and [11] into [16] gives

$$k = -\frac{\bar{F}}{\bar{y}}\,\omega^2 \exp[i\varphi] + \omega^2 m \qquad [17]$$

For a sample of thickness d and cross sectional area A, the extensional modulus $E = kd/A$. It follows from [17] that the real and imaginary parts of the complex modulus are

$$E' = -\frac{d}{A}\left\{\frac{\bar{F}}{\bar{y}}\right\}\omega^2\cos\varphi + \omega^2 m \qquad [18]$$

$$E'' = -\frac{d}{A}\left\{\frac{\bar{F}}{\bar{y}}\right\}\omega^2\sin\varphi \qquad [19]$$

In this case, the measured phase angle, φ, is different from the angle between stress and strain on the sample, δ, and so $\tan\delta \neq \tan\varphi$:

$$\eta = \tan\delta = \frac{E''}{E'} = \frac{-\dfrac{\bar{F}}{\bar{y}}\,\omega^2\sin\varphi}{-\dfrac{\bar{F}}{\bar{y}}\,\omega^2\cos\varphi + \omega^2 m} \qquad [20]$$

At low frequencies, Equation [20] reduces to $\dfrac{E''}{E'} = \tan\varphi$.

In order to calculate the inertial mass of the system to be used in Equations 18 and 20, the aluminum block is excited without the sample in a free vibration experiment. The effective mass is simply $m = F/\ddot{x}$, and as shown in Figure 11, is independent of frequency.

Figure 12, curve A, shows the storage modulus as a function of frequency as determined using Configuration II and Equation 18 for Isodamp C-1002. There is excellent agreement with the literature data (curve B) in the range 300 Hz - 6000 Hz. The loss factors calculated from Equation 20 do not compare as well to the literature data as the storage moduli do, and even after smoothing the loss factor spectrum appears noisy (Figure 13). The errors associated with this method seem to be related to errors in the phase angle at low frequencies and to the $\omega^2 m$ term at high frequencies. This method was particularly sensitive to the amount of static pre-load, with the most consistent results obtained at near zero pre-loads. Adjusting the load was sometimes difficult, since the sample was held in

place by compressing it between the aluminum block and the support, and this necessitated a pre-strain greater than the dynamic strain. An increase in the static pre-load generally resulted in increased calculated moduli.

The experimental set up for compression may be improved by using an impedance head in place of separate transducers, and a mass compensation circuit to more accurately account for the mass. The impedance head combines a force transducer and accelerometer in a single housing, allowing the measurement of impedance at the driving point of the transducer.

Configuration I was found to be a more reproducible technique than Configuration II, with better signal/noise ratio and less sensitivity to sample positioning. Also, adjustment of pre-load was more straightforward for Configuration I. The two experimental configurations described here are complementary in that they are applicable to different frequency ranges.

Conclusion

The measurement of complex Young's modulus over a range of audio frequencies was demonstrated on a commercial damping material using two variations of a forced vibration, non-resonant technique. For tensile excitation, a simple model was successful in interpreting the data up to the resonance frequency of the sample. Compressive excitation required a model that incorporated an inertial mass acting on the force transducer, but there was no interference from sample resonance. Sources of error in both cases could be traced to the phase angle, which is sensitive to resonance phenomena and low signal to noise ratio.

The method described here has several advantages over other techniques for measuring complex modulus of elastomers. First, because it is a non-resonant method and broadband excitation is used in conjunction with spectral analysis, data is available over a large range of frequencies, whereas resonance methods are limited to just a few frequencies. Second, the stiffness of the sample is directly measured, which simplifies data analysis and reduces calculation errors associated with methods that utilize transfer impedance measurements. Finally, unlike the double-lap shear method or the transfer function method, no adhesive was used in clamping the sample, which is advantageous if large numbers of samples are to be tested.

Acknowledgements

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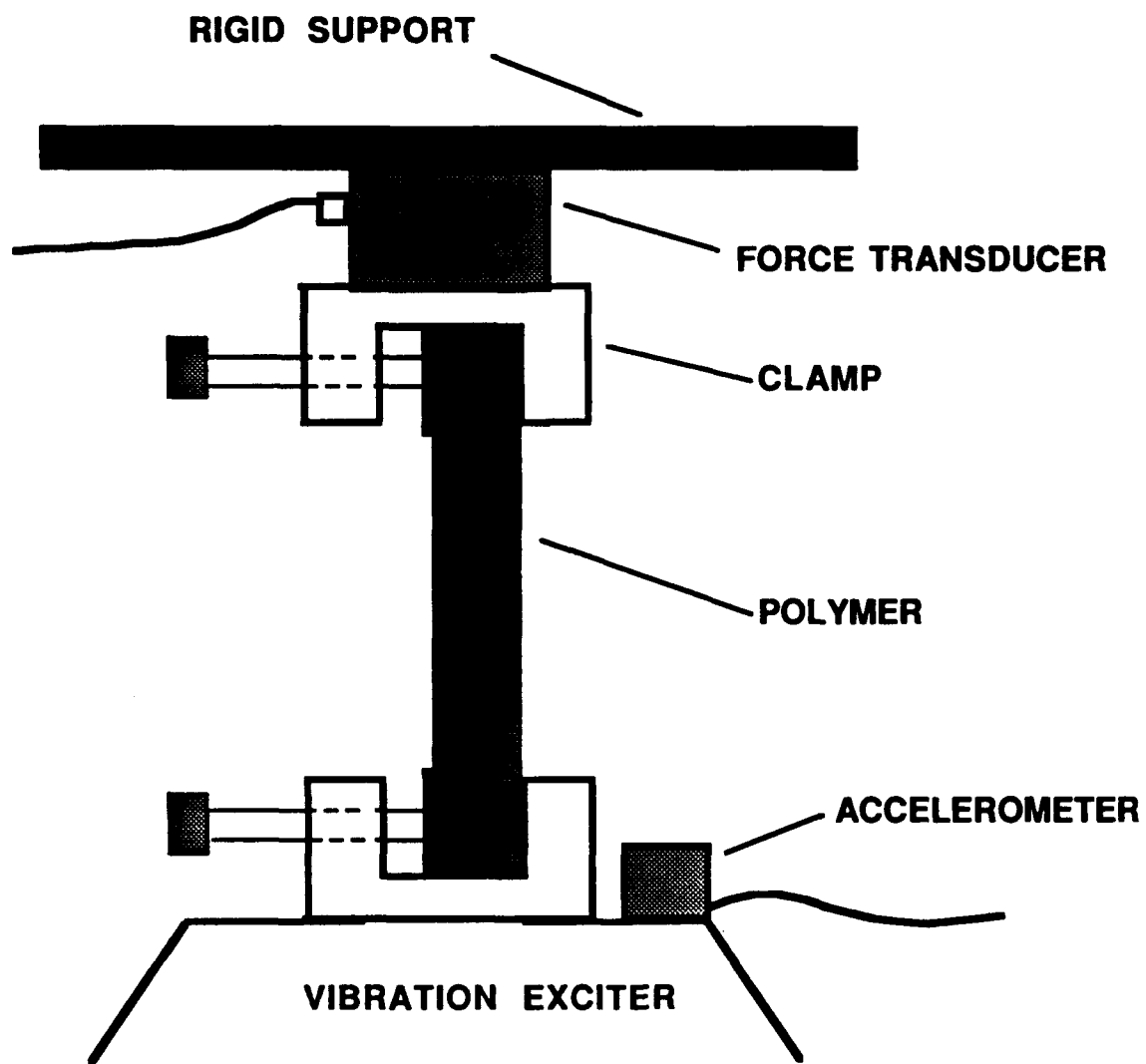
CONFIGURATION I

Figure 1. Schematic diagram of set up for tensile excitation of polymer samples.
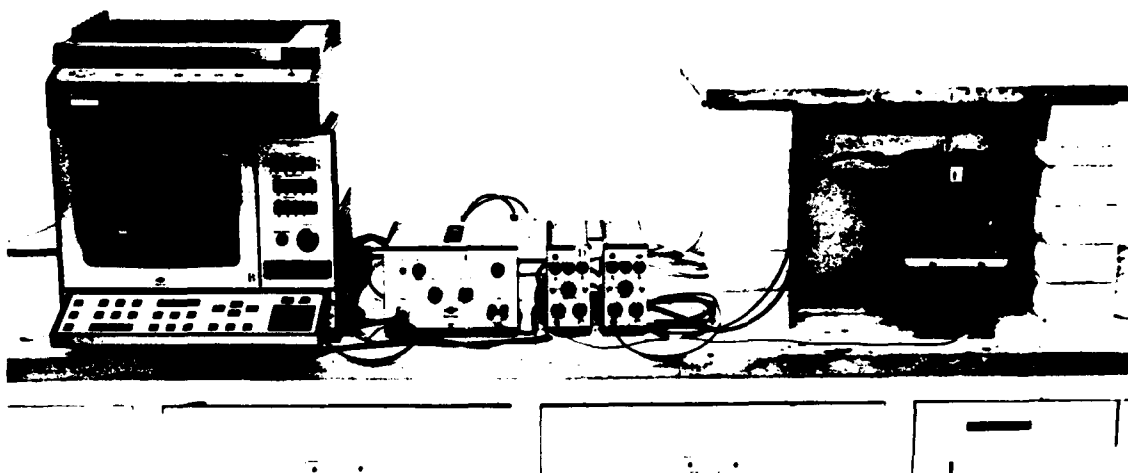
Figure 2. Photograph of experimental apparatus. (A) plotter, (B) dual channel spectrum analyzer, (C) power amplifier for vibration exciter, (D) charge amplifiers, (E) copper plate, (F) vibration exciter. The vibration exciter is supported by an adjustable lab jack.
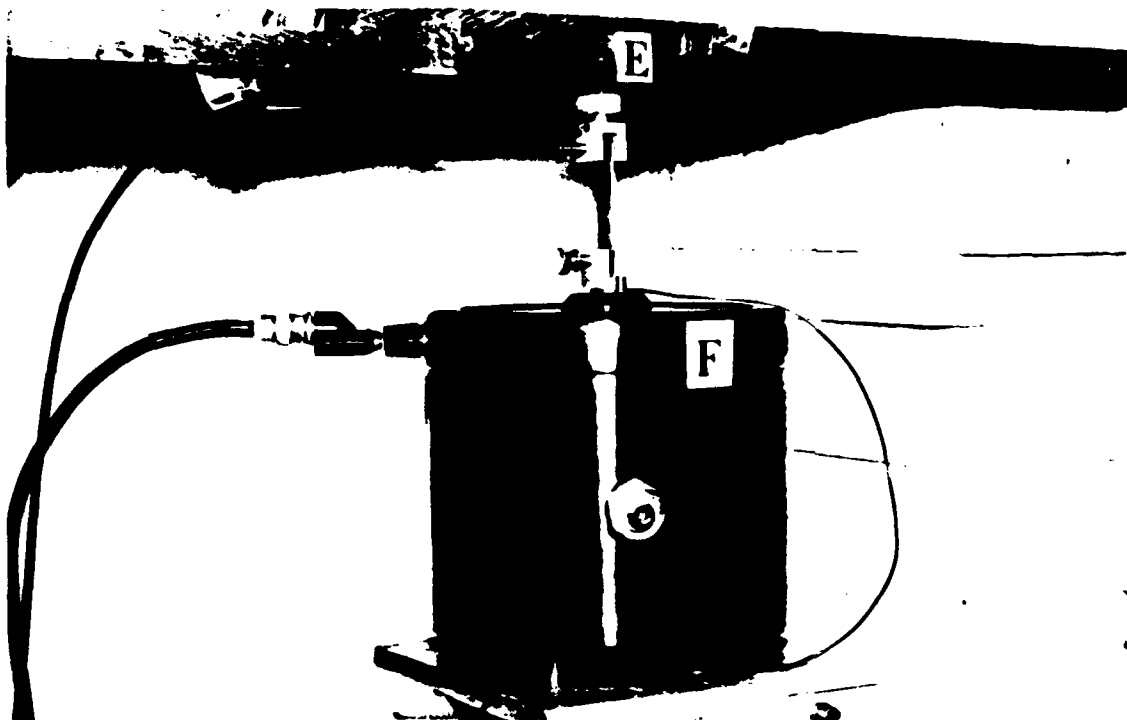
Figure 3. Close up of clamping arrangement for Configuration I. Under label E: force transducer, top clamp, sample, bottom clamp, accelerometer.
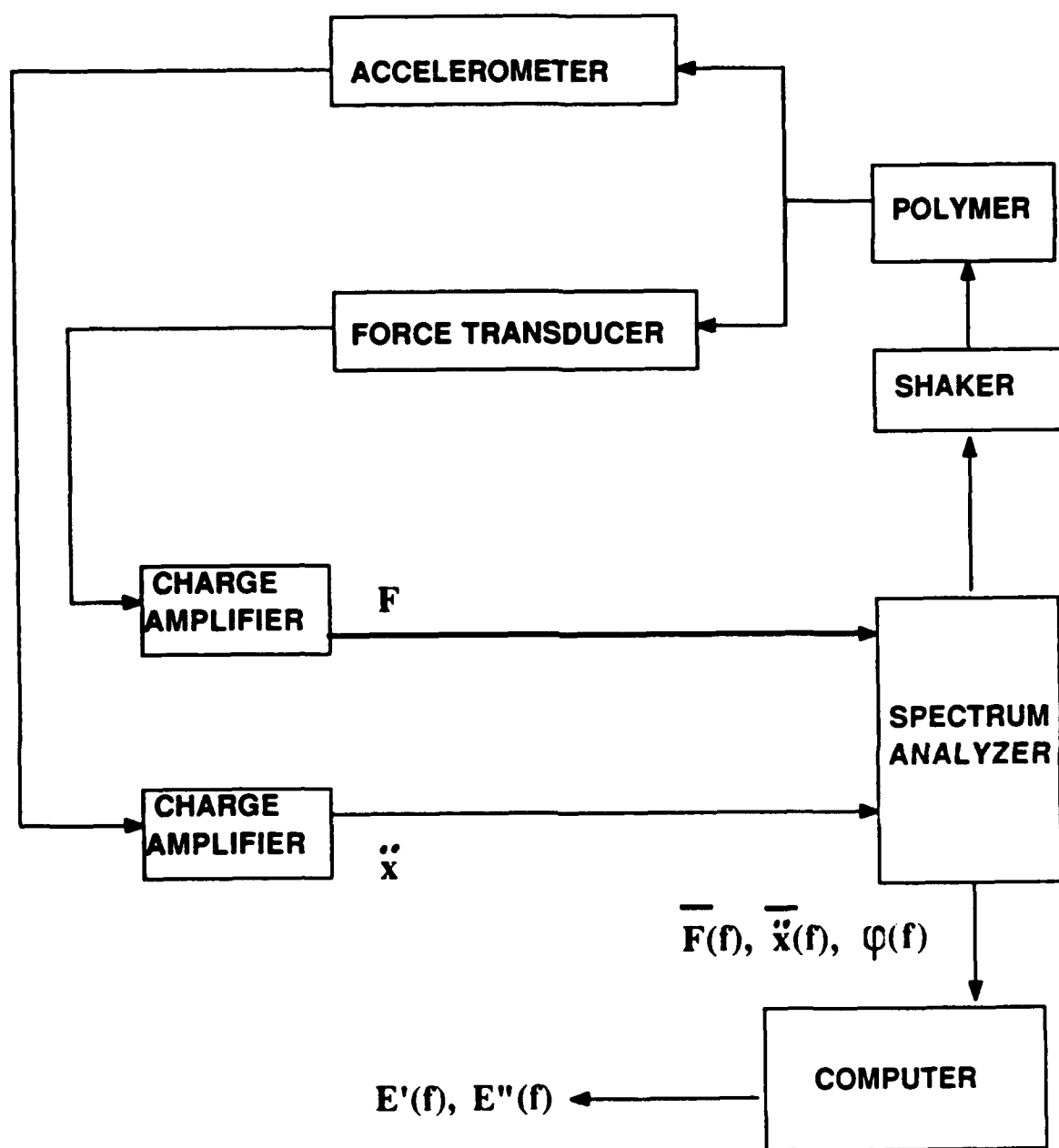
CONFIGURATION I

Figure 4. Block diagram of instruments for Configuration I.
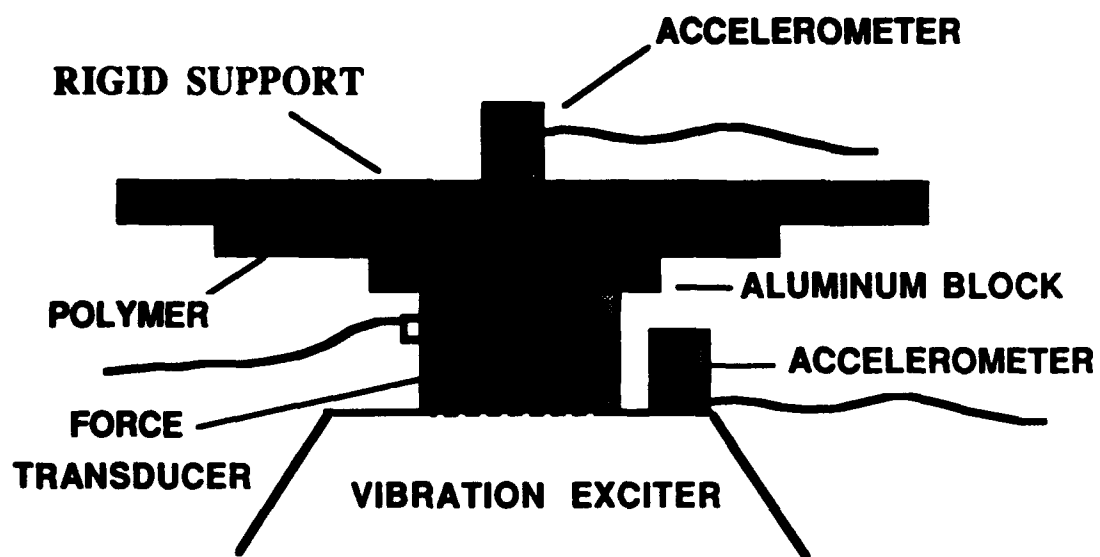
CONFIGURATION II

Figure 5. Schematic diagram of set up for compressive excitation of polymer sheets.
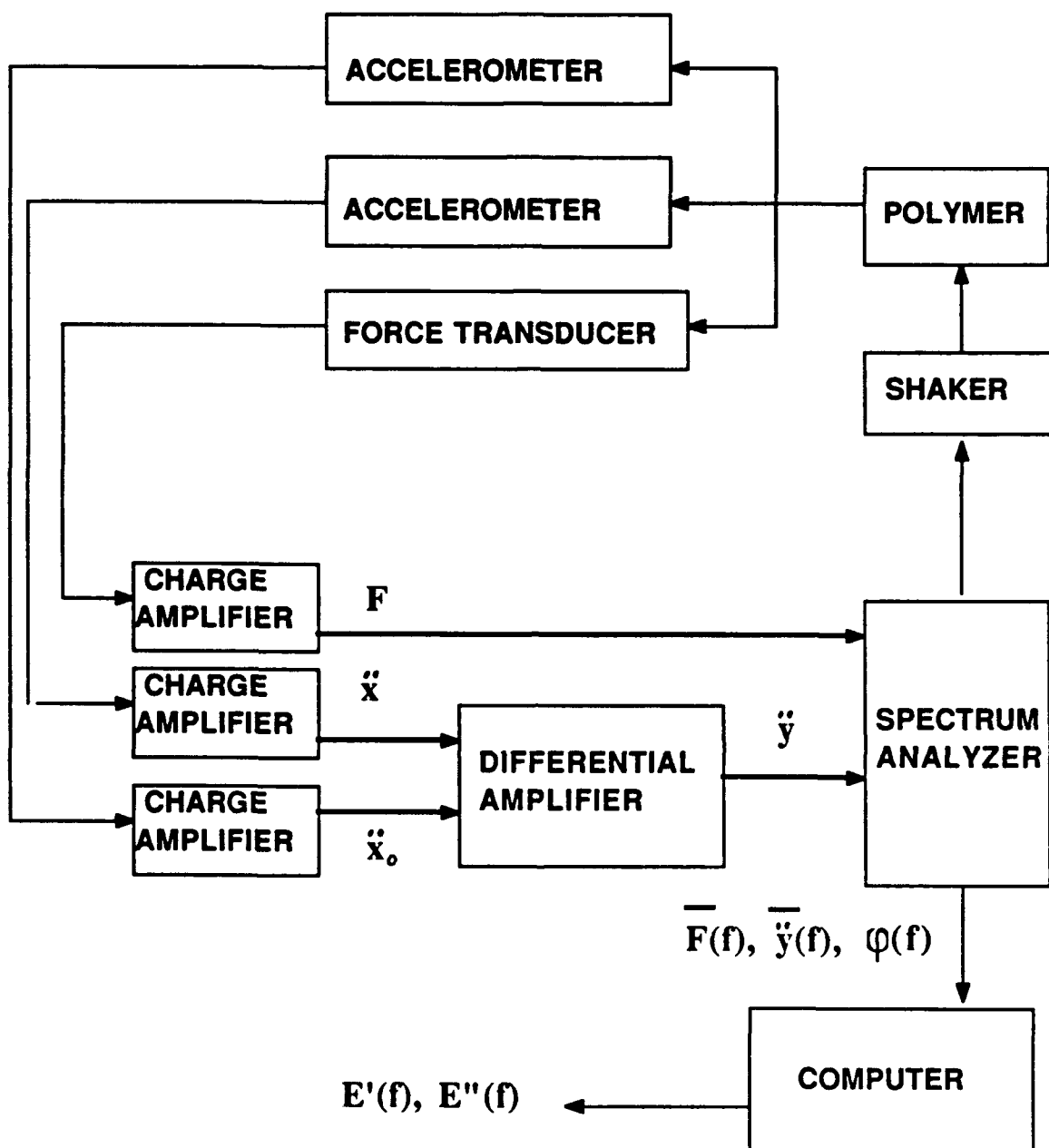
CONFIGURATION II

Figure 6. Block diagram of instruments for Configuration II.
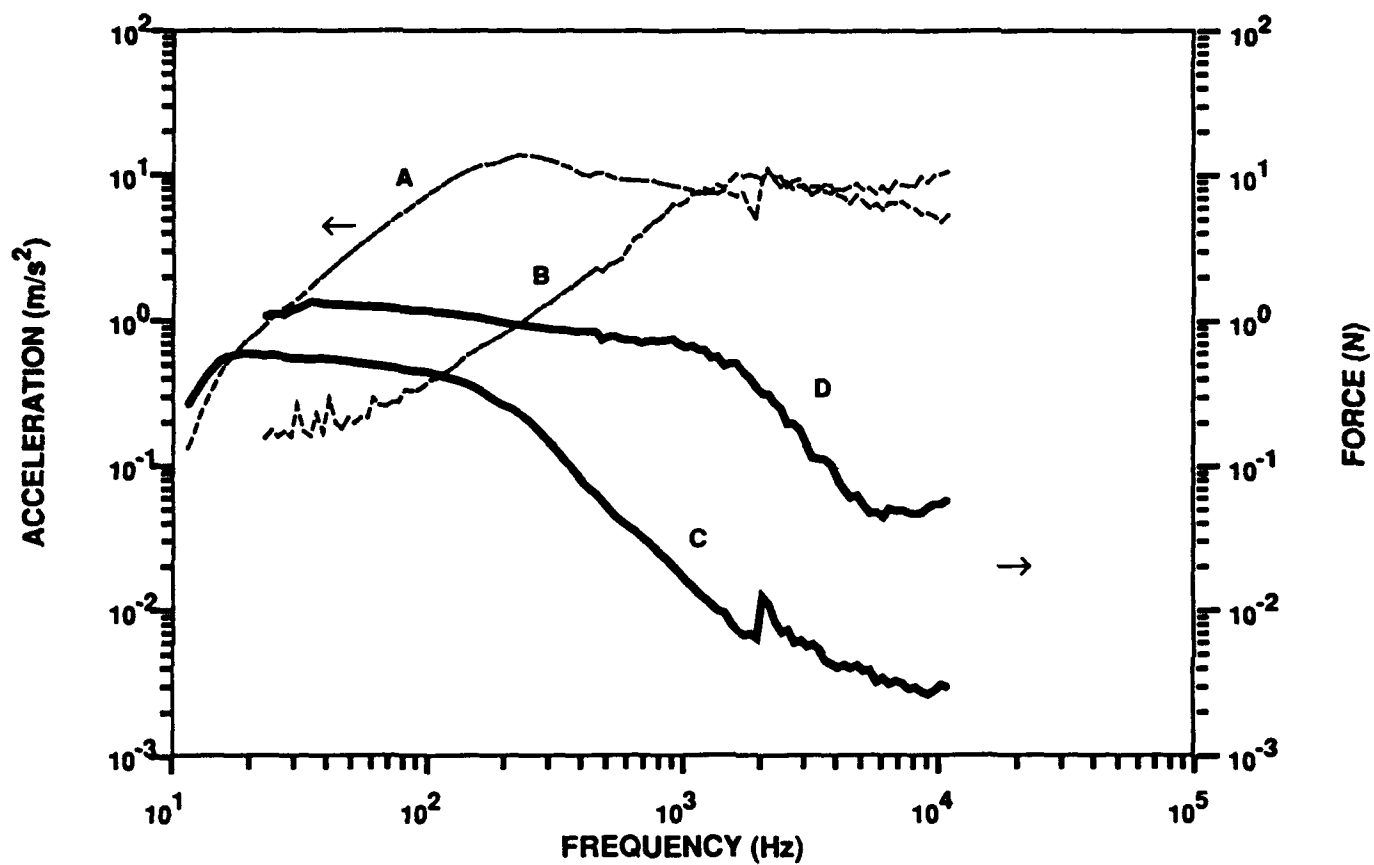
Figure 7.   Autospectra of acceleration and force signals for EAR C-1002.
(A) acceleration, Configuration I;   (B) net acceleration, Configuration II;   (C) force,
Configuration I;   (D) force, Configuration II.
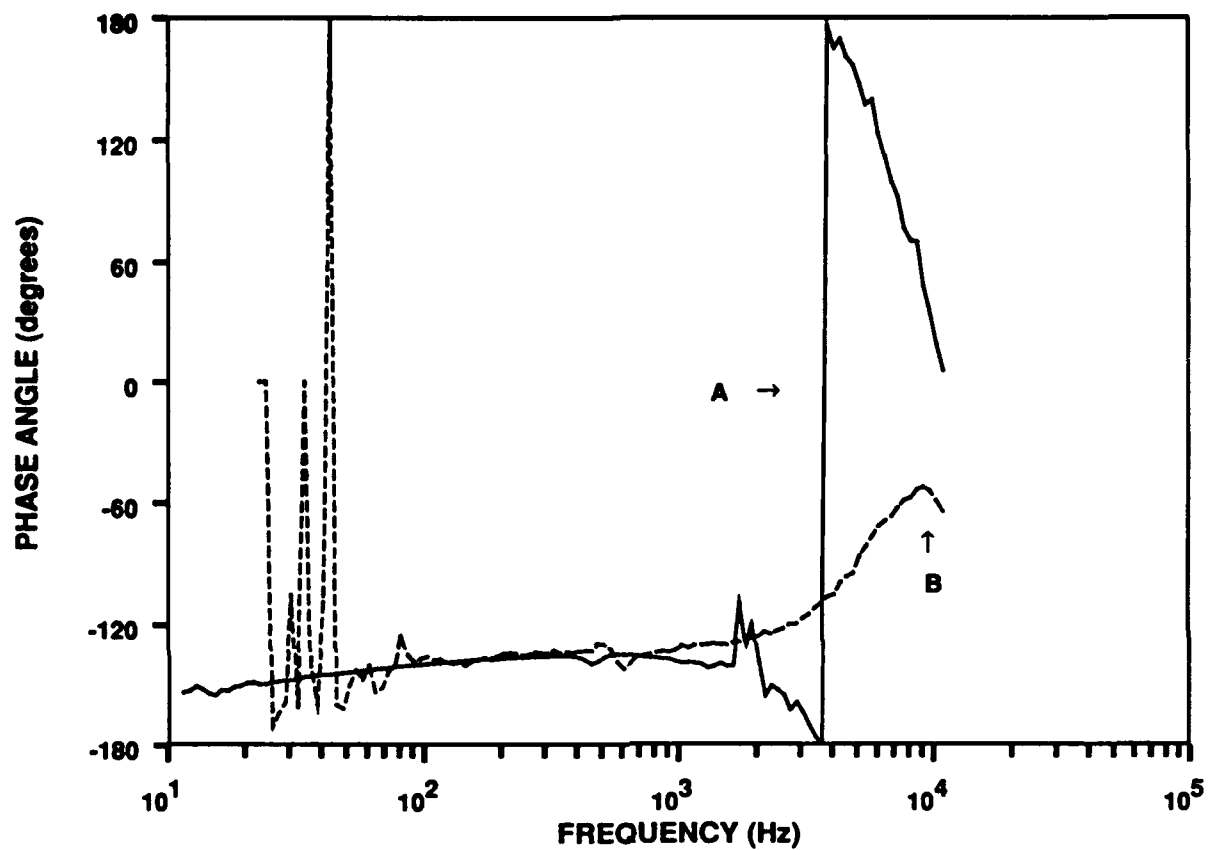
Figure 8. Phase angle between acceleration and force signals for EAR C-1002, obtained from measurement of cross spectra. (A) Configuration I and (B) Configuration II.
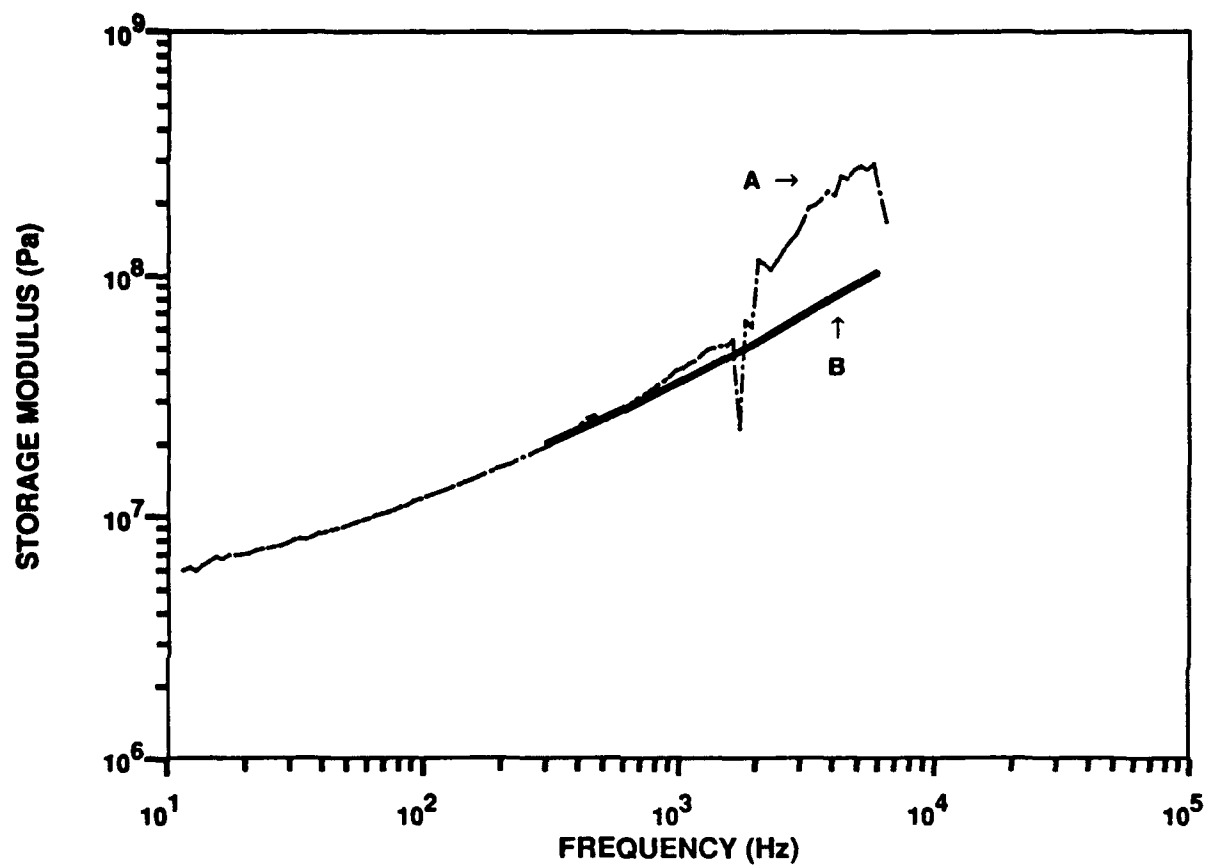
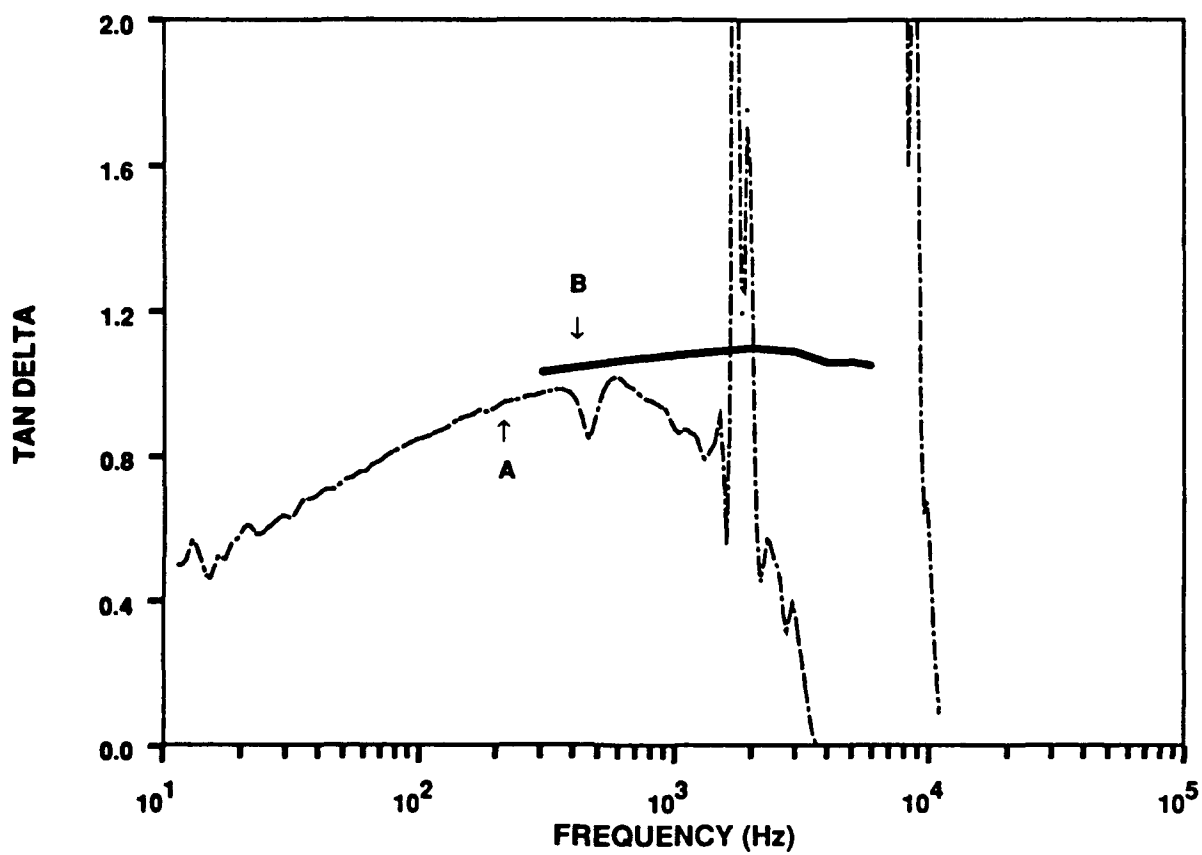Figure 9. Real part of extensional modulus versus frequency for EAR C-1002 (A) Configuration I and (B) From Reference 5.

Figure 10. Loss factor versus frequency for EAR C-1002 (A) Configuration I and (B) From Reference 5.
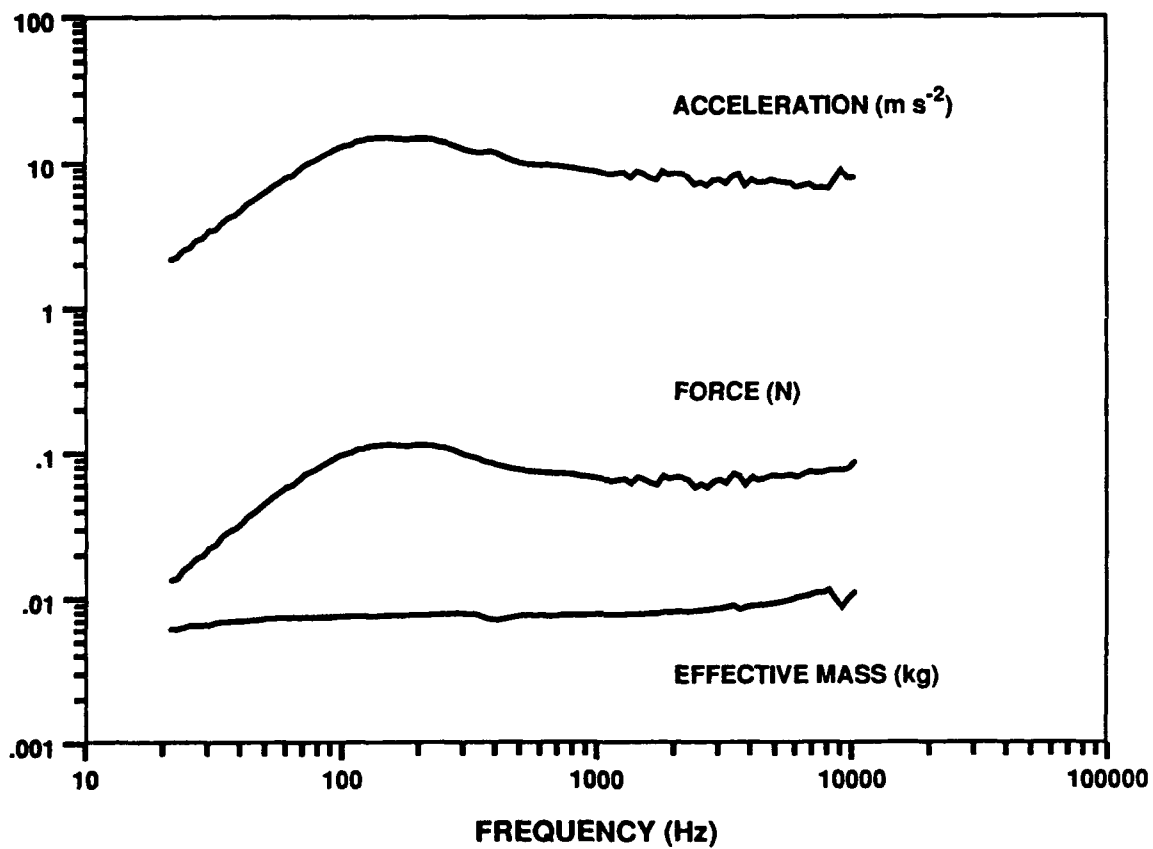
Figure 11. Plot of measured acceleration and force, and calculated mass in a free vibration experiment for estimation of inertial mass term to be used in Equations 19 - 20.
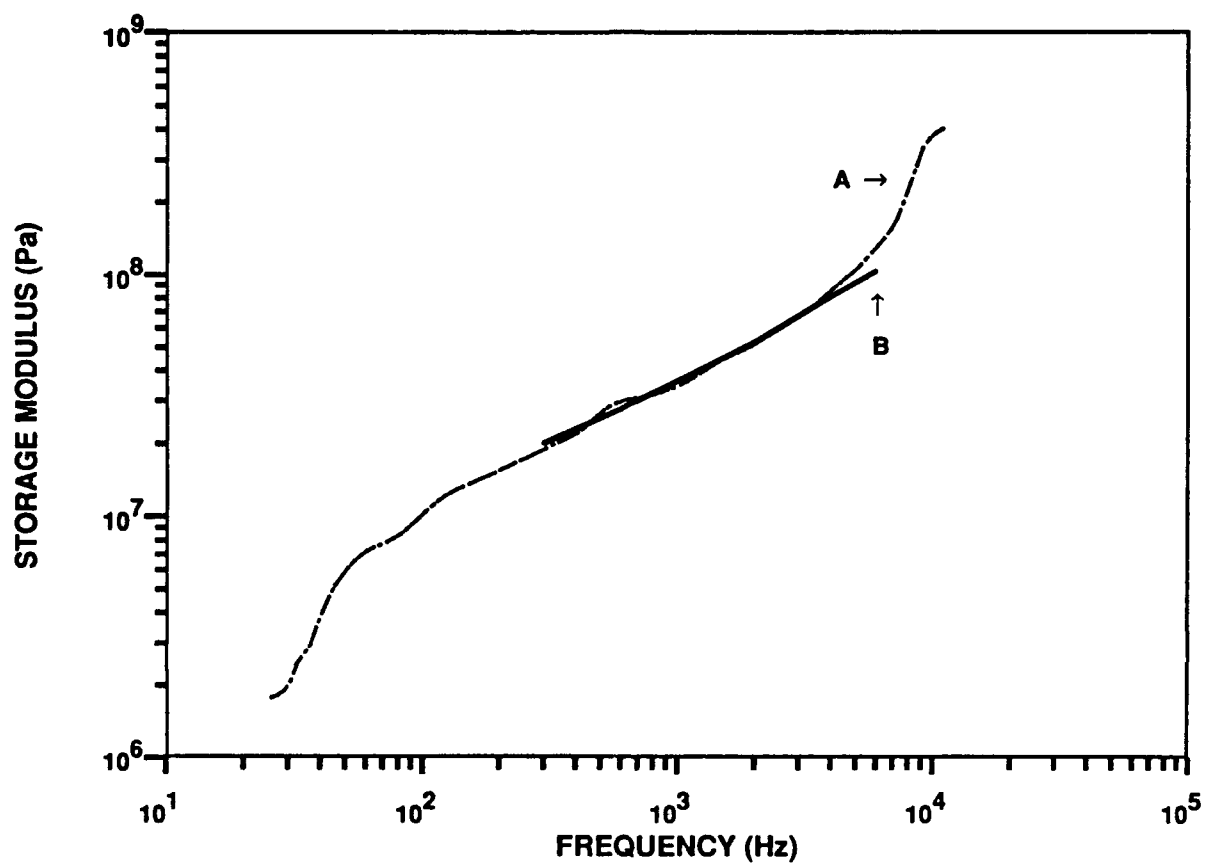
Figure 12. Real part of extensional modulus versus frequency for EAR C-1002 (A) Configuration II and (B) From Reference 5.
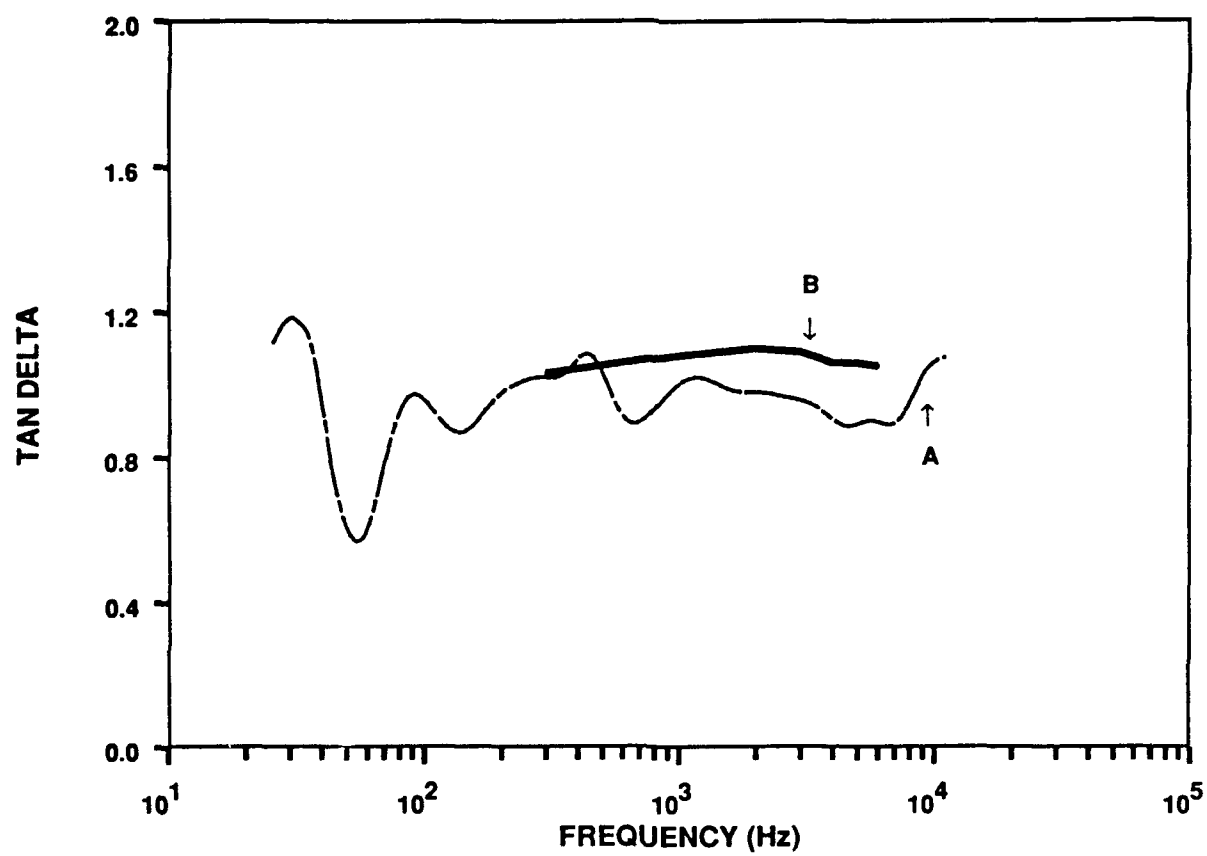
Figure 13. Loss factor versus frequency for EAR C-1002 (A) Configuration II and (B) From Reference 5.

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A forced-vibration, non-resonant method for the determination of the dynamic mechanical properties of elastomeric materials in the audio frequency range was developed and evaluated using a commercial damping material, Isodamp C-1002 from EAR Specialty Composites. Measurements of storage modulus and loss factor were made in the 11 Hz to 10 kHz range on the damping material using compressive and tensile excitation. The elastomer had a loss factor near 1.0 and a storage modulus which varied between 6×10^6 Pa and 3×10^8 Pa in the frequency range studied. For tensile excitation, the error in the calculated modulus was low up to the resonance frequency of the sample, usually ~1700 Hz. For compressive excitation of thin sheets, the error was greatest at the low and high ends of the frequency range.

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Complex modulus

Dynamic mechanical

Loss factor

Modulus

Damping material